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Manufacturing the Composition**

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(54) [Title of the Invention]

**Resin Composition and Method for
Manufacturing the Composition**

(57) [Summary]

[Object] To provide a resin composition having exceptional biodegradability as well as exceptional compatibility with other resins, melt-moldability, mechanical strength, water-repellency, and other characteristics; a method for manufacturing same; and a packaging material in which the composition is employed.

[Structure] A resin composition containing a saturated polyester resin having a hydroxy-alkanoate unit as a principal component, a vinyl alcohol polymer, and an ethylene-vinyl acetate

copolymer in a homogeneous state; and a method for manufacturing the composition. A packaging material formed from the resin composition.

[Claims]

[Claim 1] A resin composition characterized in containing (A) a saturated polyester resin having a hydroxyalkanoate unit as a principal component, (B) a vinyl alcohol polymer, and (C) an ethylene-vinyl acetate copolymer in a homogeneous state.

[Claim 2] The resin composition of Claim 1, wherein the saturated polyester resin having a hydroxyalkanoate unit as a principal component and the vinyl alcohol polymer are mixed together in a state whereby at least one of these has been mixed in advance with the ethylene-vinyl acetate copolymer.

[Claim 3] The resin composition of Claim 1, characterized in containing 32.0 to 89.1 % by weight of the saturated polyester resin (A), 8.0 to 59.4 % by weight of the vinyl alcohol polymer (B), and 1 to 20 % by weight of the ethylene-vinyl acetate copolymer (C) on a three-component basis.

[Claim 4] The resin composition of Claim 1, wherein the saturated polyester resin (A) is an aliphatic polylactone or poly- ω -hydroxyalkanoate.

[Claim 5] The resin composition of Claim 1, wherein the vinyl alcohol polymer (B) is polyvinyl alcohol or ethylene-vinyl alcohol copolymer.

[Claim 6] The resin composition of Claim 1, wherein the ethylene-vinyl acetate copolymer has a vinyl acetate unit content of 5 to 60 % by weight.

[Claim 7] A method for manufacturing a resin composition, characterized in that a saturated polyester resin having a hydroxyalkanoate unit as a principal component is blended with an ethylene-vinyl acetate copolymer, and a vinyl alcohol polymer is blended with the resulting blend.

[Claim 8] A method for manufacturing a resin composition, characterized in that a vinyl alcohol polymer is blended with an ethylene-vinyl acetate copolymer, and saturated polyester resin having a hydroxyalkanoate unit as a principal component is blended with the resulting blend.

[Claim 9] A packaging material, characterized in being formed from a resin composition containing 32.0 to 89.1 % by weight of a saturated polyester resin having a hydroxyalkanoate

unit as a principal component, 8.0 to 59.4 % by weight of a vinyl alcohol polymer, and 1 to 20 % by weight of an ethylene-vinyl acetate copolymer.

[Detailed Description of the Invention]

[0001]

[Industrial Field of Utilization] The present invention relates to a resin composition having exceptional biodegradability as well as exceptional mechanical characteristics, water-repellency, and other characteristics; a method for manufacturing the composition; and a packaging material in which the composition is employed.

[0002]

[Prior Art] The volume of solid waste effluent produced in metropolitan areas has become increasingly large over the past several years, and the limits of waste-processing performance are being tested. Plastics are consistently indicated as being one of the primary causes of such solid waste.

[0003] Degradable plastics that disappear in natural environments have attracted attention as an ideal solution for plastic wastes. Degradable plastics are classified as being either photo-degradable, wherein the polymer molecular chains break up when exposed to UV radiation, or biodegradable, wherein the plastic disintegrates through the effect of enzymes released by bacteria and fungi.

[0004] However, photodegradable plastics cannot be expected to exhibit any effect when buried in the earth, and they present risks associated with environmental pollution created by the degraded matter; therefore, biodegradable plastics are considerably more promising.

[0005] Known examples of conventional biodegradable plastics include polyhydroxybutyrate (PHB), 3-hydroxybutyrate (3HB), and 3-hydroxyvalerate (3HV).

[0006] Resin compositions formed by blending polycaprolactones (PCL) or another biodegradable plastic with another resin are already known; e.g., a biodegradable resin composition consisting of 5 to 99 % by weight of ethylene-vinyl alcohol copolymer and 95 to 1 % by weight aliphatic polyester has been cited in JP (Kokai) 4-139248.

[0007]

[Problems That the Invention Is Intended to Solve] The biodegradable plastics that are well known are stable in air, degrade in the earth and water through bacteriological action, and beneficially yield carbon dioxide gas and water as degradation products; however, they still present problems that need to be resolved in regard to the manufacture of containers, which are a prime application for plastics.

[0008] In other words, the well-known biodegradable plastics have a degradation temperature and a molding temperature that are close to each other, such as 170°C and greater and 160 to 175°C, which causes complications to occur during melt-molding even if the molded articles can be manufactured by casting methods in which chloroform or another solvent is employed.

[0009] Accordingly, when bottles or other hollow molded articles are to be manufactured, large quantities of plasticizers must be compounded with the resin to improve the melt-extrusion performance; containers so manufactured will have the undesirable effect of the plasticizers being extracted with the water (or other contents of the container). Such containers have less strength than conventional containers made from polyethylene or the like, and have hitherto only been able to exhibit the requisite strength by being made thicker.

[0010] Such biodegradable plastics are also expensive, and as such if they can be blended with another resin without their biodegradability being compromised, then dramatic advantages both in terms of melt-moldability and manufacturing cost can be expected. Other conventionally known biodegradable polymers are those that contain a hydroxyl group, but they are water-soluble, poorly water-repellent, or lacking melt-moldability, for which reason they have not been used for the above purposes.

[0011] The ethylene-vinyl acetate copolymers observed in the above prior art are melt-moldable, and have the advantage of superior water-repellency in comparison to hydroxyl group-containing polymers; however, as with the other hydroxyl group-containing polymers, they will not necessarily have adequate compatibility with aliphatic polyesters, and when fashioned into molded articles, they will undergo phase separation, suffer from mechanical cracking, fail to exhibit the anticipated resistance to gas permeability, or present other drawbacks.

[0012] The present inventors discovered that when a saturated polyester resin having a hydroxy-alkanoate unit as a principal component was blended with a vinyl alcohol polymer, and an ethylene-vinyl acetate copolymer was added to the resulting system, the compatibility among

these resin components improved dramatically along with their melt-moldability, water-repellency, and mechanical characteristics such as tensile strength and toughness. The present inventors further discovered that the blending sequence was also of great importance in terms of obtaining a blend in a uniformly dispersed state.

[0013] In other words, it is an object of the present invention to provide a resin composition having exceptional biodegradability, as well as exceptional compatibility with other resins, melt-moldability, mechanical strength, water-repellency, and other characteristics; a method for manufacturing the composition; and a packaging material in which the composition is employed.

[0014]

[Means Used to Solve the Above-Mentioned Problems] The present invention provides a resin composition characterized in containing (A) a saturated polyester resin having a hydroxyalkanoate unit as a principal component, (B) a vinyl alcohol polymer, and (C) an ethylene-vinyl acetate copolymer; and a packaging material consisting of this composition.

[0015] The present invention also provides a method for manufacturing a resin composition characterized in that a saturated polyester resin having a hydroxyalkanoate unit as a principal component is blended with an ethylene-vinyl acetate copolymer, and a vinyl alcohol polymer is blended with the resulting blend.

[0016] The present invention further provides a method for manufacturing a resin composition characterized in that a vinyl alcohol polymer is blended with an ethylene-vinyl acetate copolymer, and a saturated polyester resin having a hydroxyalkanoate unit as a principal component is blended with the resulting blend.

[0017] The resin composition used in the present invention should contain 32.0 to 89.1 % by weight of the saturated polyester resin (A), 8.0 to 59.4 % by weight of the vinyl alcohol polymer (B), and 1 to 20 % by weight of the ethylene-vinyl acetate copolymer (C) on a three-component basis.

[0018]

[Operation of the Invention] The present invention is premised on the discovery that the ethylene-vinyl acetate copolymer serves to dramatically improve the compatibility between the saturated polyester resin having a hydroxyalkanoate unit as a principal component ("saturated polyester resin" hereunder) and the vinyl alcohol polymer.

[0019] Among the resin components, it is the vinyl alcohol polymer that is the essential component for imparting biodegradability, gas permeability resistance, and other characteristics suitable for containers to the entirety of the resin composition; however, if the vinyl alcohol polymer is blended with the saturated polyester resin, virtually no compatibility will be achieved between the two components, and complications will also be presented in terms of forming a uniformly dispersed state. Furthermore, molded articles formed from the composition will have a rupture point strength that is lower than the yield point strength, as well as other serious drawbacks related to mechanical brittleness. In the present invention, when the saturated polyester resin and the vinyl alcohol polymer are to be blended, the introduction of the ethylene-vinyl acetate copolymer in advance to either of these resins can enhance compatibility and dispersibility, and eliminate the brittleness of the blend.

[0020] In the present invention, when the saturated polyester resin and the vinyl alcohol polymer are to be blended, it is important to introduce the ethylene-vinyl acetate copolymer in advance to either of these resins in order to form a homogeneous blend. FIG. 1 shows the results obtained by plotting the relation between the ethylene-vinyl acetate copolymer content and the yield point strength of molded articles formed from the resin composition. In the plot, square marks (□) show when all three components; i.e., the saturated polyester resin having a hydroxyalkanoate unit as a principal unit, the vinyl alcohol polymer, and the ethylene-vinyl acetate copolymer, have been blended together simultaneously; white circular marks (○) show when the ethylene-vinyl acetate copolymer has been blended in advance with the saturated polyester resin, and the vinyl alcohol polymer was blended with the resulting blend; and black circular marks (●) show when the ethylene-vinyl acetate copolymer has been blended in advance with the vinyl alcohol polymer, and the saturated polyester resin was blended with the resulting blend.

[0021] The results clearly illustrate the fact that in order to prevent phase separation within the blend and to improve impact resistance, it is important to avoid direct contact between the saturated polyester resin and the vinyl alcohol polymer, and to admix the ethylene-vinyl acetate copolymer with either of these resins in advance when the two are to be mixed.

[0022] This is believed to be due to the fact that the ethylene-vinyl acetate copolymer has good compatibility with the saturated polyester resin and the vinyl alcohol polymer, and will improve compatibility with the other resins and serve to prevent phase separation therein when incorporated in advance with the saturated polyester resin or vinyl alcohol polymer. As is well

known, the ethylene-vinyl acetate copolymer functions as a polymer plasticizer, and its contribution towards improving kneadability and workability as a result thereof cannot be disregarded.

[0023] In the present invention, the ethylene-vinyl acetate copolymer should be used in an amount of 1 to 20 % by weight, and especially 1 to 15 % by weight, on a three component basis. In other words, it is undesirable for the amount to be less than specified in this range, since compatibility will deteriorate, and phase separation in the molded articles will tend to occur more often. It is conversely undesirable in regard to the object of the present invention for the amount to exceed the aforesaid range, since the mechanical characteristics and gas barrier properties will deteriorate, as will biodegradability.

[0024] The saturated polyester resin should be used in an amount of 32.0 to 89.1 % by weight, and especially 50 to 80 % by weight, while the vinyl alcohol polymer should be used in an amount of 8.0 to 59.4 % by weight, and especially 15 to 45 % by weight. If the amount of the saturated polyester resin falls below the indicated range, or if the amount of the vinyl alcohol polymer exceeds the indicated range, compatibility-related problems will be presented, or biodegradability will tend to deteriorate. Conversely, if the amount of the saturated polyester resin exceeds the indicated range, or if the amount of the vinyl alcohol polymer falls below the indicated range, the gas permeability resistance and melt-moldability of the container will be inadequate.

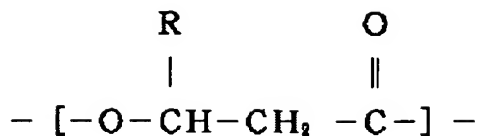
[0025]

[Preferred Embodiments of the Invention]

(Saturated polyester resin) Any biodegradable saturated polyester resin having a hydroxy-alkanoate unit as a principal component may be used as the saturated polyester resin. Such saturated polyester resins should at least have a molecular weight that will enable films to be formed, and ordinarily the number-average molecular weight thereof should be within a range of 5×10^4 to 12×10^4 , and especially 6×10^4 to 11×10^4 . Examples of preferred saturated polyester resins include poly- β -hydroxyalkanoates, aliphatic polylactones, poly- ω -hydroxyalkanoates, and copolymers of these compounds.

[0026] Examples of poly- β -hydroxyalkanoates include polymers consisting of one or more repeating units expressed by the formula below:

[Chemical Expression 1]

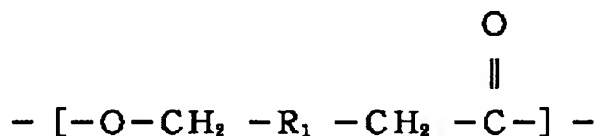


(wherein R is a straight or branched alkyl group), such as 3-hydroxybutyrate [R = -CH₃, 3HB], 3-hydroxyvalerate [R = -CH₂ CH₃, 3HV], 3-hydroxycaproate [R = -(CH₂)₂CH₃], 3-hydroxy heptanoate [R = -(CH₂)₃CH₃], 3-hydroxyoctanoate [R = -(CH₂)₄CH₃], 3-hydroxynonanoate [R = -(CH₂)₅CH₃], and 3-hydroxydecanoate [R = -(CH₂)₆CH₃].

[0027] Ideal resins of this type are copolymers formed by copolymerizing 3-hydroxybutyrate and another 3-hydroxyalkanoate, and especially 3-hydroxyvalerate. The copolymers contain these components in a weight ratio of 95:5 to 85:15 and especially 92:8 to 88:12.

[0028] Examples of aliphatic polylactones or poly- ω -hydroxyalkanoates include polymers consisting of one or more repeating units expressed by the formula below:

[Chemical Expression 2]



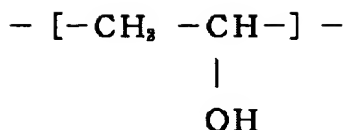
(wherein R is a straight or branched alkylene group), such as γ -butyrolactone, δ -valerolactone, and ϵ -caprolactone.

[0029] Examples of suitable types of such resins include polycaprolactones or copolymers of a caprolactone and another lactone.

[0030] Copolymers consisting of a repeating unit of Chemical Expression 1 and Chemical Expression 2 may be used in the present invention. A suitable example is a copolymer of 3-hydroxybutyrate and γ -butyrolactone or ϵ -caprolactone. Two or more of the aforescribed blends may be used.

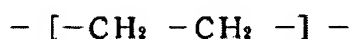
[0031] (Vinyl alcohol polymer). The vinyl alcohol polymer used in the present invention contains a vinyl alcohol unit in its molecular chain; i.e., a unit expressed by the formula below:

[Chemical Expression 3]



and ideally also contains an ethylene unit in addition to the above unit; i.e., a unit expressed by the formula below:

[Chemical Expression 4]

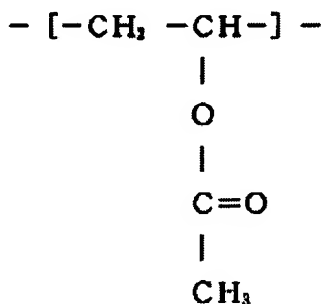


[0032] Ethylene-vinyl alcohol copolymers that are ideal in terms of melt-moldability and gas barrier properties have an ethylene content of 10 to 60 mol%, and especially 20 to 50 mol%; and have a residual vinyl ester content of 5 mol% or less, and especially 2 mol% or less. The ethylene-vinyl alcohol copolymer to be employed should have a molecular weight that enables films to be formed.

[0033] In the present invention, copolymers of a vinyl alcohol polymer or a vinyl alcohol unit and another ethylenically unsaturated monomer may be used instead of the ethylene-vinyl alcohol copolymer.

[0034] (Ethylene-vinyl acetate copolymer) Copolymers containing 5 to 60 % by weight and especially 10 to 50 % by weight of ethylene and vinyl acetate units; i.e., units expressed by the formula below, may be ideally used as the ethylene-vinyl acetate copolymer:

[Chemical Expression 5]



[0035] It is preferred in terms of the object of the present invention that the ethylene-vinyl acetate copolymer have a melt flow rate (MFR) within a range of 0.5 to 150 g/10 min, and especially 2 to 100 g/10 min.

[0036] Even if ethylene segments formed from ethylene units bonded together are present in the vinyl alcohol polymer and the ethylene-vinyl acetate copolymer used in the present invention, the molecular weight of those segments is preferably 500 or less in consideration of biodegradability. It is also preferable for segments with vinyl alcohol units bonded together to be present. With segments in which a plurality, and especially three, vinyl alcohols are bonded, the tendency is for a single secondary hydroxy group to form a keto group, and for decomposition to proceed rapidly.

[0037] (Resin composition and method for manufacturing the composition) The resin composition of the present invention contains a saturated polyester resin having a hydroxyalkanoate unit as a principal component (A), a vinyl alcohol polymer (B), and a ethylene-vinyl acetate copolymer (C); and when the saturated polyester resin and the vinyl alcohol polymer are to be blended, introducing the ethylene-vinyl acetate copolymer in advance to at least one of these resins will enable a homogeneous blend to be formed.

[0038] The ethylene-vinyl acetate copolymer may be compounded in advance with either the saturated polyester resin or the ethylene-vinyl acetate copolymer alone, or may be compounded with both resins together. In the most suitable aspect of the present invention, the saturated polyester resin having a hydroxyalkanoate unit as a principal component is blended with the ethylene-vinyl acetate copolymer, and the vinyl alcohol polymer is blended with the resulting blend.

[0039] In another preferred aspect, the vinyl alcohol polymer is blended with the ethylene-vinyl acetate copolymer, and the saturated polyester resin having a hydroxyalkanoate unit as a principal component is blended with the resulting blend.

[0040] The resin composition of the present invention may be compounded with a variety of colorants, fillers, inorganic or organic reinforcing agents, lubricants, plasticizers, leveling agents, surfactants, viscosity improvers, viscosity reducers, stabilizers, antioxidants, UV ray absorbers, rust inhibitors, or the like depending on the application.

[0041] According to the present invention, the saturated polyester resin (A) or the vinyl alcohol polymer (B) and the ethylene-vinyl acetate copolymer (C) are melted, kneaded, and blended with the remaining resin components; and the resulting blend is kneaded, extruded, or injected to manufacture a packaging material. In the present invention, the blend can be applied to packaging materials that consist of only one layer, but can also be used to form packaged articles

in the configuration of a multilayered structure laminated with other resin layers as long as the blend forms a continuous layer. Examples of well-known lamination techniques that may be used therefor include co-extrusion, co-injection, extrusion coating, sandwich lamination, and dry lamination.

[0042] When packaging materials or the like are manufactured, a mixture composed of one of the resin components and a blend consisting of the ethylene-vinyl acetate copolymer and the other resin component is supplied to the hopper of an extruder or injection machine. Both components of the mixture may be either dry-blended or melt-blended. Dry-blending may be performed by using a ribbon blender, conical blender, Henschel mixer, or any of a variety of other mixers, while melt-blending may be performed by using a single- or twin-screw extruder, kneader, Banbury mixer, roll, or other apparatus. The fact that dry-blending may also be used to provide overall operational convenience is a characteristic of the present invention.

Manufacturing the blend with the ethylene-vinyl acetate copolymer should be performed in the same manner as with melt-blending.

[0043] The extruder is preferably any that is provided with a screw. Flat or ring dies may be used, and T-die or inflation methods may be used to form films or the like. Hollow-molding the extruded parison enables bottles, tubes, tanks, or other hollow containers to be molded.

[0044] Any injection machine known to be provided with an injection plunger or a screw may be used, and the aforescribed mixture is injected into the injection mold via a nozzle, sprue, and gate. The resin is thereby introduced into the injection mold cavity and cooled to a solid to yield the packaging material of the present invention.

[0045] (Applications) The composition of the present invention may be used for bottles, cups, tubes, plastic cans, pouches, caps, and a variety of other plastic packaging containers; films, trays, or other packaging materials; containers, tanks, baskets, and other circulating containers; and pipes, cases, and other structures.

[0046]

[Working Examples] The present invention shall be described in detail below with reference to working examples.

[0047] (Molding method) Pellets were formed by melt-blending using a TEM35B twin-screw extruder (manufactured by Toshiba Machine) with an extrusion temperature of 180°C and a

screw rotation rate of 100 ppm [sic]. The same conditions were also used when melt-blending was performed in two steps. The pellets were extruded into 0.5 mm-thick sheets.

[0048] The pellets were subjected to direct blow molding using an ES65 extruder (manufactured by Ikegai) at an extrusion temperature of 180°C, a screw rotation rate of 300 rpm, a mold temperature of 16°C, and a blown air pressure of 6 kg/cm².

[0049] (Measuring method) Tensile strength measurements were performed at 500 mm/min using a Tensilon UCT-5T manufactured by Orientec, with a load cell of 100 kg. An ASTM D-1822 die was used for the shape of the samples. The results for mechanical strength were obtained from these measurements.

[0050] Working Example 1

Polycaprolactone H-7 (A) manufactured by Daicel Chemical Industries was used as the poly- ω -hydroxy alkanooate for the saturated polyester resin, Eval G (B) manufactured by Kuraray was used for the vinyl alcohol polymer, and Evatate R5011 manufactured by Sumitomo Chemical was used for the ethylene-vinyl acetate copolymer.

[0051] The saturated polyester resin (A), vinyl alcohol polymer (B), and ethylene-vinyl acetate copolymer (C) were melt-blended in the following weight ranges to obtain a molded sheet, which was used to show the relation between the ethylene-vinyl acetate copolymer content and the mechanical strength (yield point strength) in FIG. 1.

(A):(B):(C) = 69.5:29.5:1 to 60:25:15

[0052] The molded sheet readily tended to rupture, which was presumed to be due to the flow of the vinyl alcohol polymer being oriented in the MD direction; therefore, the mechanical strength results shown were obtained with a measurement sample of the molded sheet punched out in the TD direction.

[0053] In FIG. 1, two types of blends obtained using different sequences; i.e., one blend was obtained by melt-blending resin (A) and resin (C), and then blending resin (B) with the resulting blend, and the other blend was obtained by melt-blending resin (B) and resin (C), and then blending resin (A) with the resulting blend, were compared with a blend formed by melt-blending resin (A), (B), and (C) together in one process. Strength was noticed to increase when the ethylene-vinyl acetate copolymer (C) content was in the 1 to 15 % by weight range, and the optimal strength characteristics were obtained when the content ratio in the molded article was 5 % by weight.

[0054] As shown in Table 1, the change in mechanical strength, which drew attention to the composition ratio between the saturated polyester resin (A) and vinyl alcohol polymer (B) in the three-component resin composition, follows the increase in the vinyl alcohol polymer content. Since the sheet ruptured and was brittle, as was presumed to be due to the flow of the vinyl alcohol polymer being oriented in the MD direction, the composition ratio between the saturated polyester resin (A), vinyl alcohol polymer (B), and ethylene-vinyl acetate copolymer (C) were adjusted to the following range:

$$(A):(B):(C) = (32.0 \text{ to } 89.1):(8.0 \text{ to } 59.4):(1.0 \text{ to } 20.0)$$

[0055] Working Example 2

The saturated polyester resin (A) and vinyl alcohol polymer (B) as well as the ethylene-vinyl acetate copolymer (C) were blended in a weight composition ratio of (A):(B):(C) = 67:28:5 according to the procedure cited in the section describing the molding method, with the resulting blend being subjected to direct blow-molding. The resulting bottle-shaped article had a volume of 400 mL and a flattened thickness of 0.4 mm. The article had the same strength characteristics as that of the article described in Working Example 1, and exhibited rigidity but no brittleness. Ten of the articles were filled with distilled water that had a temperature of 2°C, sealed tightly, and dropped from a height of 1.2 m, but none of them broke.

[0056]

[Table 1]

Saturated polyester resin (A)/ vinyl alcohol polymer (B)/ ethylene-vinyl acetate copolymer (C)		93:2:5	90:5:5	66:29:5	38:57:5	35:60:5
Mechanical strength	MD	△	○	○	○	△
	TD	△	○	○	△	×
Rigidity		×	○	○	○	×

- Suitability for use as a container: completely satisfactory
- △ Suitability for use as a container: somewhat satisfactory
- × Suitability for use as a container: unsatisfactory

[0057]

[Effect of the Invention] According to the present invention, an ethylene-vinyl acetate copolymer is blended with a saturated polyester resin having a hydroxyalkanoate unit as a principal component or a vinyl alcohol polymer, and the resulting blend is blended with the remaining resin or a resin composition to yield a resin composition having exceptional biodegradability as well as excellent resin compatibility, melt-moldability, mechanical strength,

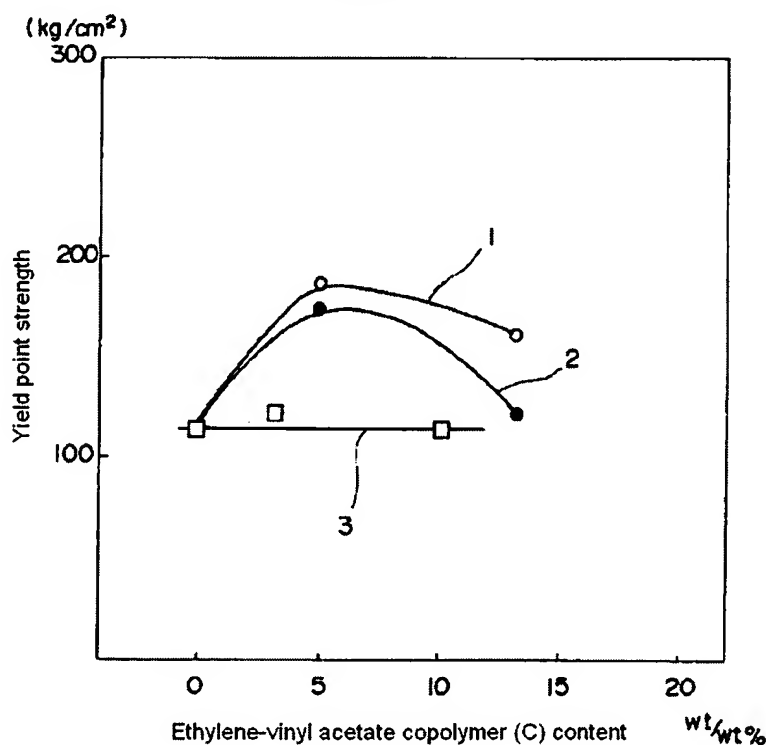
water-repellency, and other characteristics. The use of the composition to manufacture packaging materials enables exceptional benefits to be realized.

[Brief Description of the Drawings]

[Figure 1] Graph showing the ethylene-vinyl acetate copolymer (C) content in Working Example 1 and the yield point strength relating to the method for molding the blend. The curves in the graph refer to values obtained with the following samples:

- (1) A molded sheet obtained by melt-blending the saturated polyester resin (A) with the ethylene-vinyl acetate copolymer (C), and blending the vinyl alcohol polymer (B) with the resulting blend.
- (2) A molded sheet obtained by melt-blending the vinyl alcohol polymer (B) with the ethylene-vinyl acetate copolymer (C), and blending a saturated polyester resin (A) with the resulting blend.
- (3) A molded sheet obtained by blending together the saturated polyester resin (A), vinyl alcohol polymer (B), and ethylene-vinyl acetate copolymer (C) in one process.

[Figure 1]



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